



Heat Chambers for DiffusIR™ Accessory

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General Information

Technical Support

Technical information describing the use and theory of diffuse reflectance and its sampling options are available from PIKE Technologies. Additionally, customer support and answers to your questions are also available from PIKE Technologies at 608-274-2721

The Manual

This manual is designed to be informative and easy-to-use regardless of your experience with FT-IR sampling accessories. It is recommended you familiarize yourself with the installation and the temperature controller before using it.

Packing & Unpacking

Upon arrival, please check the carton to ensure that all pieces have been received and that no pieces are damaged. We suggest that you save the carton for future shipment if required.

General Product Information

Descriptions:

This device is a variable temperature cell for use with the DiffusIR diffuse reflectance accessory. The sample can be heated from room temperature up to 500° or 900° within the chamber. Any temperature can be set in this range and can be controlled automatically with the controller. The environment within the chamber can be pressurized up to 100 atmospheres if you have the high pressure (HP) option.

Features

- z Any temperature can be set from room, up to the temperature limit of 500° or 900°.
- z This Heat Chamber has been designed for use with the DiffusIR accessory from PIKE Technologies. This accessory has a wide sampling area which allows the use of a large capacity heater and which, in turn, dramatically improves the durability of the heater.
- z Vacuum achievable: 1×10^{-6} Torr (13×10^{-4} Pa)
- z Maximum Pressure with HP option: 100 atmospheres
- z Ceramic sample cups included
- z Inserting and changing samples is easily accomplished by opening and closing the screw cap which holds the window.
- z Wiring is self-contained within the body of the Heat Chamber for ease of use.
- z A coolant line can be attached quickly using the unique “One-Touch” mechanism.
- z A larger 1/8 inch gas tubing line enables pressure to be reduced quickly.
- z The entire optical housing can be purged, thus enabling measurements to be made without unwanted water vapor.

Technical Specifications

Specification:

- Temperature range : Room Temp to 500° or 900°
- Thermocouple : K Type
- Reachable Vacuum : 1×10^{-6} Torr (13×10^{-4} Pa)
- Maximum Pressure : 100 Atmospheres (tested with N₂ gas)
with high pressure adaption option
- Leaking volume : Less than 6.0×10^{-11} Pa. m³/sec
- Sample Cup Type: Ceramic, porous base
- Sample Cup Size : 6.0 mm OD, 4.0 mm height
4.7 mm ID, 2.0 mm depth
- Maximum Heater Voltage : Approx. 28 V 3A 84W
- Accuracy of temp control : ± 0.5 %
- Cooling port : Mini Auto Joint (One-Touch)
O.D. 6mm I.D. 4mm Synflex Tube
- Gas/Vacuum Port : 1/8" Swage Lock
- Window : KBr 32x3 mm
- Dome Optic : ZnSe dome included with HP option

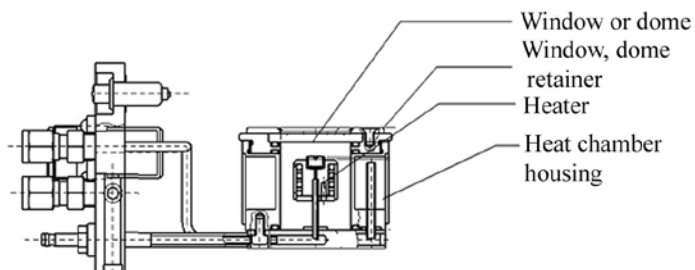
Parts List:

Heat Chambers 500° or 900° Versions, with Optional High Pressure Adaption

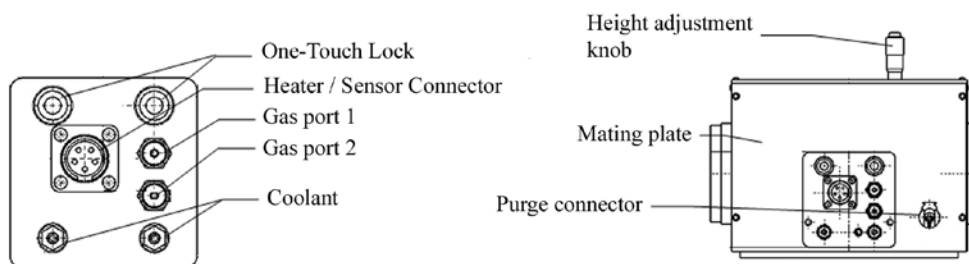
Description	Remarks	Qt
Heat Chamber main frame	Window: w/ plug 1/8" Swage lock with Ferrule and nut	1
HP chamber adaption	Optional	0 or 1
Mating Plate	Replaces DiffusIR front panel	1
KBr Window	32 x 3 mm	1
Dome Crystal	ZnSe, included with HP option only	1
Sample Cups / Ceramic	Porous base for gas permeation	3
Alignment Disk	Fits in sample cup	1
Auto Join (Female)	Joint for coolant	2
Wrench	Screw with flat head in M3	1
Wrench	For M3CAP	1
Coolant tube	6-4 mm L = 2M Black	2
Sample Press Stick		1
Coil tube	SUS304 1/8" L = 1M	2
Plug valve	SUS 1/8" Swagelok	2
O-ring Extra	Viton S-25	2
O-ring Extra	Viton S-3	2

Technical Specifications

Description of parts:

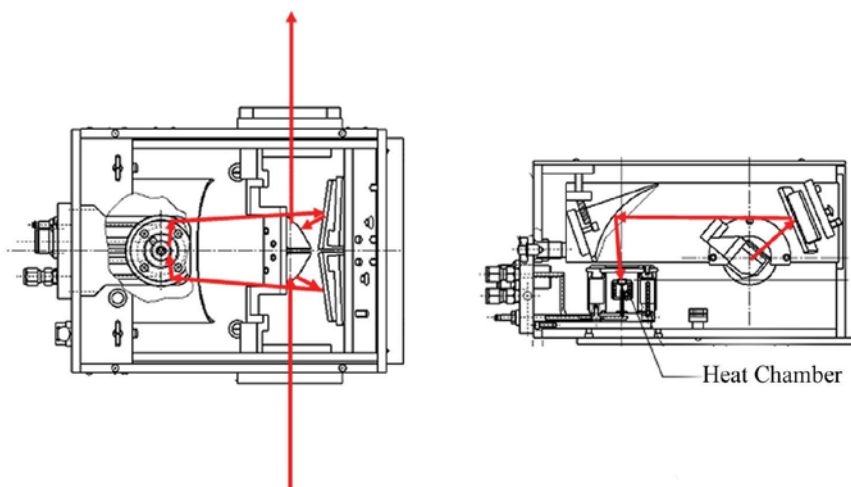


Heat Chamber, side view

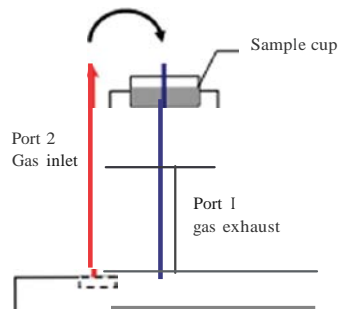
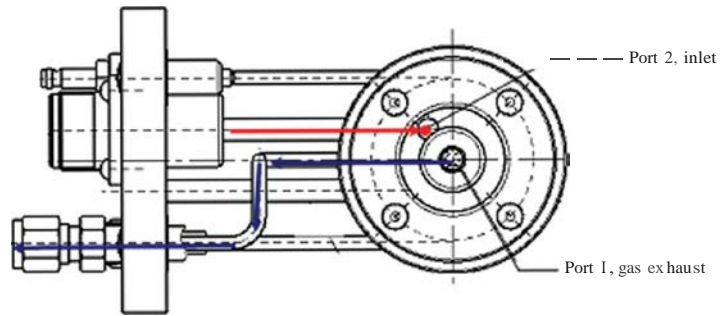
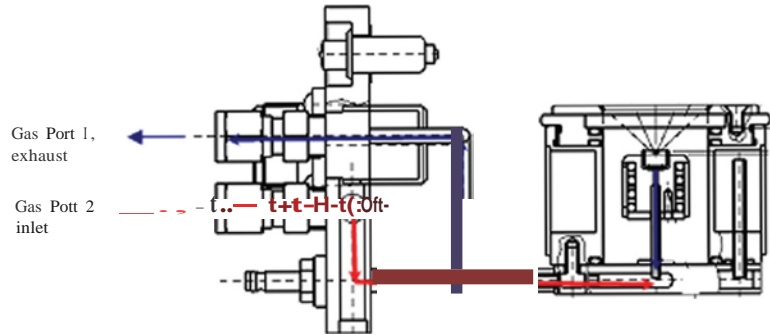


Heat Chamber, DiffusIR Connections

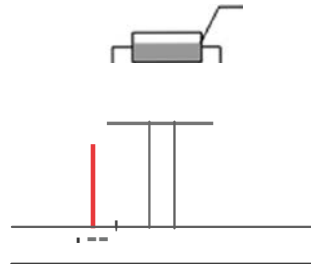
Optical Layout:



Gas flow



When you want to flow through the sample, gas input is Port 2 and gas exhaust is Port 1 underneath the sample cup.



If you evacuate the chamber through Port 2, the sample may be disturbed or scattered out of the sample cup.

Installation

1. Remove the standard front panel and standard sample stage from the DiffusIR. The screws for the standard sample stage are found at the base of the DiffusIR accessory.



2. Remove the gas port for purging from the plate and attach it to the mating plate of the heat chamber. Then, mount the gas port via the mating plate to the DiffusIR.

3. For the heat chamber without HP, remove the window cover of the heat chamber by unscrewing with your fingers. For the heat chamber with HP adaption, remove the 8 large screws holding the domed cover with the ZnSe crystal.

Do not remove the small screws – this may cause failure of the pressure seal of the cell.



4. Place the alignment mirror on the sampling area of the chamber. Mount the heat chamber to DiffusIR via the mating plate. Always ensure to set the height adjustment knob to its maximum height of 5mm before inserting the chamber. Mount it to the mating plate on the front of the DiffusIR using the two One-Touch Locks so that the chamber is properly attached to the mating plate. These Locks will detach and attach easily by pressing on it with your finger.



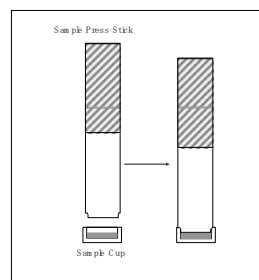
5. The DiffusIR should next be re-aligned. Using the adjusting height knob, maximize the throughput energy. For better alignment of the DiffusIR, please refer to the DiffusIR User Manual for maximizing throughput energy.

6. Switching between the HP and not HP versions of the chamber is easily done by removing the 4 screws at the under side base of the chamber and installing the other version. Be sure to have the gaskets installed when you make this change.

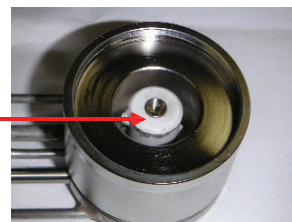
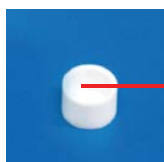
Using the Chamber

1. Remove Heat Chamber from DiffusIR and remove the chamber cover exposing the heater.

2. Load the sample into the ceramic sample cup. The ceramic sample cups have a porous base and may be used with all sampling modes including use of reaction gases. When the sample is loaded, the sample should be set at 1mm lower than sample surface by using Sample Press Stick.



3. Set the sample cup into the heater.



4. Replace the chamber cover by screwing it back into position or replacing the large screws and mount the Heat Chamber into the DiffusIR.



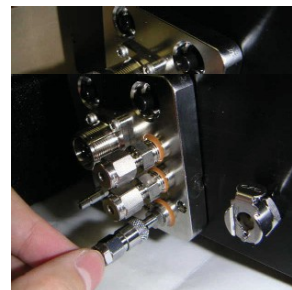
Note: If any sample should spill into the Heat Chamber, remove the Dome from the chamber using the four screws located at the bottom of the chamber. Then, clean device. If the o-ring has also had sample spilled on it, please be sure to clean it.

5. The gas lines should next be attached.

6. Next connect the temperature controller to the Heat Chamber using the circular connector.

7. The Power cable should be connected to the temperature controller.

8. Plug the power cable to an AC outlet.



9. Attach the connection portion of the tube which is 4 mm I.D. to the coolant joint. This has to be done just one time. After that, attaching and detaching of coolant tube can be done using the quick-connect feature of this fitting.

10. Mount the coolant joint to Heat Chamber and to permit water to flow. To detach, grasp the outside of the joint with your fingers and press the joint towards the body of the Heat Chamber.

11. When the Heat Chamber is mounted to DiffusIR, usually the One-Touch lock is used.

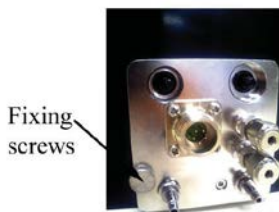
* Besides the One-Touch Lock, if user wants to mount it tightly with screws, please follow the instruction below:

- a) Remove the guide pins from DiffusIR Panel.



- b) Mount the HC to DiffusIR using One-Touch lock as it is without guide pin.

- c) For fixed operation, substitute Fixing Screws for guide pins.



Replacement Parts

Here is a list of replacement parts for use with the heat chambers for the DiffusIR accessory;

160-1132	32 x 3 mm KBr disk
160-1113	32 x 3 mm ZnSe disk
160-1159	32 x 3 mm Si disk
162-4210	O-Ring, DiffusIR HC, (10 ea.)
162-4215	O-Ring, DiffusIR HC Cooling Line, (10 ea.)
162-4251	DiffusIR HC Porous Ceramic Cup, (1 ea.)

Precautions

Temperature will be controlled within the dome in a vacuum environment between room temperature and the chamber limit of either 500° or 900°.

Depending on the flow rate, temperature and type of atmospheric gas, the temperature

may not go up to the chamber limit temperature. Especially with Helium or Hydrogen gas where the thermal conductivity is high, the temperature can not reach to that level.

Examples of the maximum achievable temperatures at the flow of following gases for the 900° heat chamber:

N₂ Gas 100cc/min 700°

He Gas 100cc/min 550°

When the Heat Chamber is heated up, cooling water must be used.

a) Pressure range of coolant : More than 0.1kgf/cm² and less than 5kgf/cm²

b) Temperature of coolant : 6° ~28°

c) Flow rate of coolant : 50ml/min as recommended

• The pressure and flow rate of the gas :

Flow rate : Maximum 1L/min (Recommended)

Pressure : Maximum 1kg/cm²

Precautions specific to the High Pressure adaption for the Heat Chambers.

Depending on flow rate, temperature and type of atmospheric gas, the temperature might not go up to 900 deg C. Especially when used with Helium or Hydrogen gas where the thermal conductivity is high. The temperature will not reach the maximum specified. Please see chart below.

Guidelines for the maximum temperature under pressure

(Conditions: N₂ Gas flow 100cc/min /900 C, N₂ gas pressure (kgf/cm²) (N₂ gas purity 99.9%)

• 10 (kgf/cm ²)	/710°
• 20	/630°
• 30	/580°
• 40	/540°
• 50	/510°
• 60	/490°
• 70	/450°
• 80	/430°
• 90	/410°
• 100	/400°

* When the chamber is heated over the above temperatures, the life time of the heater will be reduced.

Guidelines for the maximum temperature with flow of following gases:

N₂ Gas 100cc/min 700 °

He Gas 100cc/min 550 °

*The numerical values mentioned above are approximate.

*The test of pressure and vacuum was done using N₂ gas.

When it is heated up, cooling water must be used.

- a) Pressure of coolant : Between 0.1kgf/cm² and 5kgf/cm²
- b) Temperature of coolant : 6 deg C ~28 deg C
- c) Flow rate of coolant : 50ml/min as recommended

Other precautions

- a) Do not loosen the small screws of the dome.
 - b) Periodically inspect the O-ring. If there is a crack or signs of stress, please replace it.
 - c) Before removing the jacket, purge the water out of the cooling line
 - d) Use caution when removing/replacing jacket to not touch the heater housing.
- The pressure and flow rate of the gas :
Flow rate : Maximum 1L/min
Pressure : Maximum 1kg/cm²